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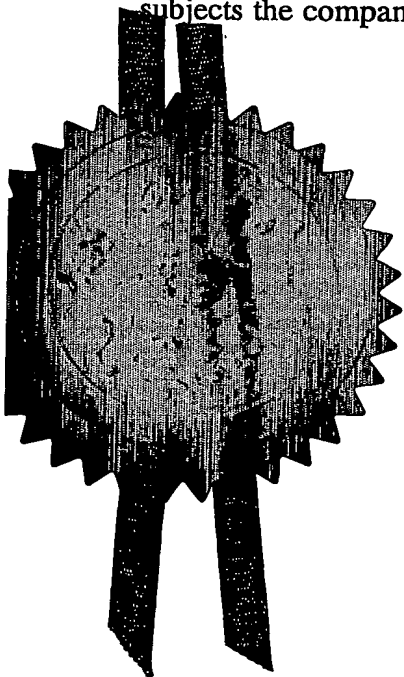
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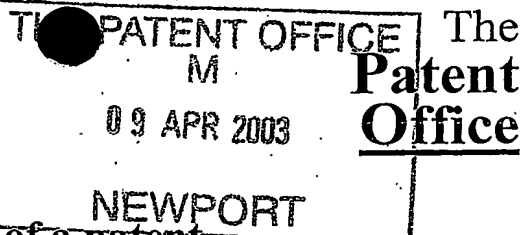
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1.	Your reference	JPP202		
2.	Patent application number (The Patent Office will fill in this part)	03081353		
3.	Full name, address and postcode of the or of each applicant (underline all surnames)	BAC2 Ltd 27 Stubbington Way Fair Oak Eastleigh Hants SO50 7LQ United Kingdom (GB)		
	Patents ADP number (if you know it)	8353344001		
	If the applicant is a corporate body, give the country/state of its incorporation	United Kingdom		
4.	Title of the invention	Solid Polymer Electrolyte		
5.	Name of your agent (if you have one)	Barker Brettell		
	"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)	10-12 Priests Bridge LONDON SW15 5JE		
	Patents ADP number (if you know it)	7442494003		
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7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application		Date of filing (day/month/year)
8.	Is a statement of inventorship and of right to grant of a patent required in support of this request (Answer 'Yes' if: a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an applicant, or c) any named applicant is a corporate body. See note (d)).	YES		

# Patents Form 1/77

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Description 15

Claim(s)

Abstract

Drawing(s) 1 + 1

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination  
(*Patents Form 9/77*)

Request for substantive examination  
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1. I/We request the grant of a patent on the basis of this application.

Signature

*Barker Brettell*

Date

Barker Brettell

08 April 2003

2. Name and daytime telephone number of person to contact in the United Kingdom

James P. Peel

Tel: 020 8392 2234

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## SOLID POLYMER ELECTROLYTE

The present invention relates to a solid polymer electrolyte important in electronic applications such as the manufacture of fuel cells and batteries.

5

Solid electrolytes are desirable in the construction of cells, batteries and capacitors because the problem of leakage from liquid electrolytes is avoided. Solid polymer electrolytes also offer other advantages over liquid electrolytes in that they enable the fabrication of cheaper, safer, smaller and lighter solid state products which are suitable for use in many different applications.

10

Typically, solid polymer electrolytes are prepared from mixtures of polymers, non-volatile solvents and alkali metal salts. Polymers such as poly(alkylene oxide), poly(vinyl fluoride), poly(vinylidene fluoride), polysulfone, polyacrylonitrile, polyester, polyether, poly(ethylene)imine, polymethacrylate, poly(ethylene succinate), poly(N-propylaziridine), poly(alkylene sulphide)s, poly(ethylene adipate), or copolymers are known. Solid polymer electrolytes are made most practically by dissolving the solid polymer and salts in high boiling point polar organic solvents prior to the casting of thin films.

15

20

For solid polymer electrolytes to exhibit high ionic conductivity the salt must dissolve in the polymer matrix. It is also essential that the solid electrolytes have sufficient mechanical and dimensional stability as for example, in the manufacture of batteries the main objective is to obtain a solid electrolyte to separate the two electrodes. It is also important that they are chemically stable towards electrodes and other components in the polymer electrolyte systems.

25

30

A major drawback with the commonly employed polymers is that they tend to be crystalline and therefore the ionic conductivity decreases when the temperature drops below the melting point of the matrix polymer. For example, the complex polymer film consisting of polyethylene oxide and alkali metal salt has an ionic conductivity of around  $10^{-3}$  S cm<sup>-1</sup> at temperatures of 100°C, or higher but an ionic conductivity of  $10^{-8}$  S cm<sup>-1</sup> at room temperatures. The solid polymer electrolyte is therefore limited to high temperature operating conditions. This low conductivity at ambient temperatures is recognised as a major limitation for widespread application of solid polymer electrolytes.

A solution to these problems has been sought.

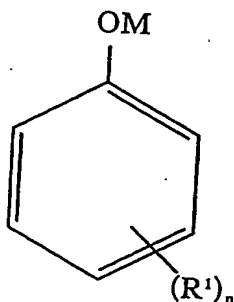
According to the invention there is provided an electrical device including two asymmetric electrodes and an ester-cured alkaline phenolic resole resin containing conducting alkaline salts.

It has surprisingly been found that the electrolyte used in the invention is not crystalline in its solid state and exhibits good conductivity at ambient temperature. Without intending to be bound by any supposed theory, it is believed that the cured composition is useful as an electrolyte because dissolved alkaline salts, essential for conductivity, are formed in-situ during the curing process, thereby negating the need to prepare a salt polymer solution.

Cured phenolic resins are thermoset polymers and are superior to all other resin systems with respect to their good thermal and mechanical stability, and their flame resistance. Normally they have good electrical insulating capabilities too. It is therefore surprising that an ester-cured alkaline resole resin is useful as an electrolyte.

The electrical device according to the invention is preferably a cell, a battery including two or more cells, or a capacitor (especially an electrolytic capacitor).

- 5 The ester-cured salt-containing alkaline phenolic resole resin is preferably a reaction product of an ester curing agent with a phenolic resole and a base. The phenolic resole is preferably a reaction product of a phenol-reactive aldehyde with an alkaline compound of formula



- wherein  $R^1$  is a straight or branched chain optionally unsaturated alkyl group containing from 1 to 8 carbon atoms (preferably from 1 to 4 carbon atoms, more preferably from 1 to 2 carbon atoms) optionally substituted by a halogen atom (preferably chlorine) or a hydroxy group, a
- 20 halogen atom (preferably chlorine), a hydroxy group, and/or a phenyl or benzyl group (optionally substituted by a hydroxy group and/or a straight or branched chain alkyl group containing from 1 to 8 carbon atoms (preferably from 1 to 4 carbon atoms, more preferably from 1 to 2 carbon atoms) optionally substituted by a halogen atom (preferably chlorine) or a
- 25 hydroxy group);

- $M$  is a mixture of hydrogen ions and at least one further cation (preferably the at least one further cation is an alkali metal cation (preferably sodium, lithium or potassium), an alkaline earth metal cation (preferably barium, magnesium or calcium), and/or a  $N(R^2)_4^+$  ion
- 30 (wherein each  $R^2$  is the same or different and is hydrogen or a straight or branched chain alkyl group containing from 1 to 4 carbon atoms)) wherein

the molar ratio of hydrogen ions to the at least one further cation is sufficient for the pH to be greater than 7 and is preferably from 2:1 to 1:1; and

n is from 0 to 2.

5

Examples of suitable compounds of formula (I) include, but are not restricted to the salt of phenol itself, salts of substituted phenols such as alkylated phenols, halogenated phenols and polyhydric phenols, and hydroxy-substituted poly-nuclear aromatics. Examples of alkylated phenols include methylphenol (also known as cresol), dimethylphenol (also known as xlenol), 2-ethylphenol, pentylphenol and tert-butyl phenol. Examples of halogenated phenols are chlorophenol and bromophenol. Examples of polyhydric phenols include 1,3-benzenediol (also known as resorcinol), 1,2-benzenediol (also known as pyrocatechol), 1,4-benzenediol (also known as hydroquinone), 1,2,3-benzenetriol (also known as pyrogallol), 1,3,5-benzenetriol and 4-tert-butyl-1,2-benzenediol (also known as tert-butyl catechol). Examples of hydroxy-substituted poly-nuclear aromatics include 4,4'-isopropylidenebisphenol (also known as bisphenol A), 4,4'-methylidenebisphenol (also known as bisphenol F) and naphthol.

20

Salts of compounds formed by the condensation reaction of two or more compounds of formula (I) with one or more molecules of a phenol-reactive aldehyde are suitable for use in the ester-cured alkaline resole resin. Examples include, but are not limited to, resinous reaction products of phenol itself, salts of substituted phenols such as alkylated phenols, halogenated phenols and multi-hydroxy phenols, and hydroxy-substituted multi-ring aromatics. Furthermore, mixtures of aldehyde-reactive phenols, such as those obtained from coal tar fractionation, depolymerised lignin and cashew nut shell liquid, can be employed as all or part of the resole component.

25

30

The phenol-reactive aldehyde used to react with the compound of formula (I) to form an alkaline phenolic resole is preferably a compound of formula

5



(II)

wherein R represents a hydrogen atom or a straight or branched chain alkyl group having from 1 to 8 (preferably from 1 to 4, more preferably from 1 to 2, most preferably 1) carbon atoms; or a precursor of a compound of formula (II).

Examples of suitable aldehydes include formaldehyde, acetaldehyde, propionaldehyde, n-butylaldehyde, n-valeraldehyde, caproaldehyde. Compounds suitable for use as precursors for a compound of formula (II) include compounds that decompose to formaldehyde such as paraformaldehyde, trioxane, furfural, hexamethylenetriamine, acetals that liberate formaldehyde on heating, and benzaldehyde.

The aldehyde is preferably reacted with the compound of formula (I) in a ratio of from 1:1 to 1:3, preferably from 1:1.2 to 1:3, more preferably from 1:1.5 to 1:3.

Some of the alkalis  $\text{M(OH)}_x$  (where M represents a non-hydrogen cation and x represents 1 or 2) are not very soluble in an aqueous resin e.g. calcium hydroxide. They can still be used by dehydrating the resin and using the ester as a solvent for the resole. The water insoluble alkali can then be dispersed in the resin to form a paste. A polar solvent (for example water) is then required to start the reaction.

30



The ester curing agent used to cure the alkaline phenolic resole resin is preferably of formula



(III)

5

wherein  $R^3$  represents a hydrogen atom or a straight or branched chain alkyl group containing from 1 to 8 carbon atoms (preferably from 1 to 4, more preferably from 1 to 2 carbon atoms) optionally substituted by a halogen atom; and

10  $R^4$  represents a straight or branched chain alkyl group containing from 1 to 8 carbon atoms (preferably from 1 to 4, more preferably from 1 to 2 carbon atoms) optionally substituted by one or more hydroxy and/or  $R^3COO^-$  groups, or

15 a phenyl group optionally substituted by a straight or branched chain optionally unsaturated alkyl group containing from 1 to 8 carbon atoms (preferably from 1 to 4 carbon atoms, more preferably from 1 to 2 carbon atoms) optionally substituted by a hydroxy group, a halogen atom (preferably chlorine), a hydroxy group, and/or a phenyl or benzyl group (optionally substituted by a hydroxy group and/or a straight or branched

20 chain alkyl group containing from 1 to 8 carbon atoms (preferably from 1 to 4 carbon atoms, more preferably from 1 to 2 carbon atoms)); or

$R^3$  represents a chemical bond to  $R^4$  and  $R^4$  represents a straight or branched chain alkyl group containing from 2 to 10 carbon atoms (preferably from 2 to 4 carbon atoms).

25

The ester curing agent for the alkaline resole resin must be reactive with the alkali to produce a salt in the cured resin. Reactive esters can include, but are not restricted to, carboxylic acid esters, esters of polyhydric alcohols, lactones and carbonate esters, phenolic esters and

30 resole esters. Examples of reactive carboxylic acid esters are methyl formate and ethyl formate. Examples of reactive polyhydric alcohol esters

which may be used as curing agent for the resin include glycerol triacetate and ethylene glycol diacetate. Examples of reactive carbonate esters include cyclic carbonate esters such as propylene carbonate and ethylene carbonate. Examples of reactive lactones include propiolactone, butyrolactone, valerolactone and caprolactone. Examples of reactive phenolic esters are phenyl acetate and resorcinol diacetate. An example of a reactive resole ester is 2,4,6-*tris*-acetoxymethylphenyl acetate. Mixtures of esters may be used for example propylene carbonate and triacetin to vary the rate of cure.

10

It has been found that the rate of curing of the resole by the ester is determined primarily by the acidity of the conjugate acid, e.g. ethyl formate ( $R^3 = H$ ) reacts approximately 1000 times faster than ethyl acetate ( $R^3 = CH_3$ ) due to the greater acidity of formic acid over acetic acid. The gel times achieved using each ester also exhibit a similar order of difference. The carbon chain length of the alcohol ( $R^4$ ) influences saponification rates and gel times to a lesser extent with the saponification rate reduced and the gel time increased with each additional carbon. It is also been found that as the chain length and/or branching of  $R^3$  and  $R^4$  increases the miscibility of the resin and ester is reduced. Good compatibility between the ester and resin is essential for the cure reaction to proceed. It is therefore clear that the selection of an ester curing agent will determine the cure rate of the reaction, and will also determine the carboxylate ion formed in the reaction and ultimately the salt contained in the polymer matrix which influence the properties of the electrolyte.

25

The base used in the reaction to form the ester-cured salt-containing alkaline phenolic resole resin used in the invention is preferably an alkaline compound which is capable of forming a conducting salt which is soluble in the resin used in the invention. An example of a suitable base is a hydroxide or an oxide of an alkali or alkaline earth metal or of

30

ammonia, e.g. lithium, sodium, potassium, magnesium, calcium, barium or ammonia.

5 The ester-cured salt-containing alkaline phenolic resole resin used in the invention is prepared from a resole resin, an ester curing agent, one or more bases and, optionally, a polar solvent. Methods for their synthesis are well known to a person of skill in the art and are described in DE-C-1 065 605, DE-C-1 171 606, JP-A 49-16793 and JP-A 50-130627. According to these publications, a highly alkaline phenolic resole resin in  
10 aqueous solution may be cured at ambient temperature by reaction with an organic ester by contacting the resin with the ester in the form of a liquid or a gas. The ester-cured alkaline phenolic resole resin containing conducting alkaline salts used in the invention is optionally in dried or in anhydrous form depending on the particular properties required.

15 Ester cured alkaline phenolic resoles can be differentiated from acid cured resoles in that the polymer matrix of the cured alkaline phenolic composition contains a high level of alkaline salts. To disperse or dissolve salts of carboxylic acids is very difficult in liquid resoles as the  
20 phenolic resin can lose solubility and precipitate from solution. Secondly, acidifying a phenolic resole containing dispersed salts of carboxylic acids will generate CO<sub>2</sub> due to the decomposition of the salt on reaction with the acid. It is therefore a unique feature of the cured alkaline resole composition that high quantities of carboxylic acid salts  
25 are present in the cured polymer matrix having been formed during the cure reaction.

The resin used in the invention optionally includes a plasticiser to increase flexibility of the resin. It will be appreciated that for some  
30 applications a flexible resin would be useful. The plasticiser is preferably inert, alkali compatible, non-volatile, and/or liquid. Preferably the

plasticiser is soluble in the resin and/or the ester curing agent. Preferably the plasticiser is present in an amount from 5 to 30% by weight, more preferably from 10 to 20% by weight. Examples of the plasticiser include an excess of the ester curing agent, polyvinylacetate and/or a polyethylene glycol.

The nature of the asymmetric electrodes used in the electrical device according to the invention will depend upon the nature of the salt in the electrolyte. It will be straightforward for a person of skill in the art to determine what electrodes are to be used. Examples of suitable electrodes include a zinc negative electrode and a positive electrode made of materials selected from the group of silver or a nickel/molybdenum/chromium alloy wherein the polymer electrolyte is in contact with the negative and positive electrodes and contains dissolved alkaline salts; or a lithium-ion cell using a positive electrode of cobalt oxide and carbon in the form of graphite or coke as the negative electrode where the electrolyte contains a dissolved lithium salt.

The invention also provides use of an ester-cured salt-containing alkaline resole resin as an electrolyte or as an electromagnetic shielding material or to prevent electrostatic discharge at a location. Examples of applications for the resin used in the invention as an electromagnetic shielding material include housings for electronic products, such as computers, cash registers, portable phones and other consumer electronics, anti-static packaging materials for use with electronic components or with fine powders, e.g. foods, where there is a risk of dust explosions caused by electrostatic discharge.

The invention further provides a method of suppressing electromagnetic interference in a product which method includes shielding the product with an ester-cured salt-containing alkaline resole resin. The shielding

preferably involves providing the product with a housing constructed at least partially from the ester-cured salt-containing alkaline resole resin. The product may be an electrical or electronic product.

- 5 The invention also provides a method of preventing electrostatic discharge at a location which method includes providing an ester-cured salt-containing alkaline resole resin at the location. A suitable location might be a packaging for a fine powder or for an electronic device or component such as a micro chip or a printed circuit board; or the location might be a  
10 floor covering, a gas meter part, a water pump seal or a self lubricating bearing; or a workbench or a similar location where devices or components sensitive to electrostatic discharge are manipulated or otherwise worked on.
- 15 The invention is illustrated by way of example with reference to the following drawing:

Figure 1 shows an outline cross-sectional view of an electrical device according to the invention.

20

- Figure 1 shows an electrical device 1 which has a block of electrolyte 2 with asymmetric electrodes 3a,3b at each side of it. The asymmetric electrodes are each provided with electrical connectors 4a,4b. The electrodes are asymmetric electrodes in that the two electrodes do not  
25 have the same composition such that they are made from different materials. Examples of suitable asymmetric electrodes are given above but they are not intended to limit the scope of the claims.

- As an alternative to the illustrated embodiment, the electrical connectors  
30 4a,4b could be replaced by current collectors as is commonly known in the art.

The following examples illustrate how to prepare an electrolyte for use in the invention. The benefits of the invention are also demonstrated. In these examples the materials used are a conventional alkaline phenolic resole (resin A), a highly alkaline resole (resin B), a neutral aqueous resole (Resin C), butyrolactone and triacetin (esters), para-toluene sulphonic acid (catalyst), polyethylene glycol (plasticiser) and graphite (conductivity promoter).

#### EXAMPLE 1

##### Resin A: Preparation of an alkaline resole

Resin A is an alkaline phenol-formaldehyde resin with a formaldehyde to phenol molar ratio of 2.0:1 and a sodium hydroxide to phenol molar ratio of 0.65:1. Phenol (5.0 mol) and sodium hydroxide (0.1 mol) were charged to a reaction vessel and the temperature maintained at 65°C whilst 50% formalin (3.0 mol) was added. The temperature was allowed to be raised to 80°C and maintained at 80°C while a second charge of 50% formalin (5.0 mol) was added slowly over 30 minutes. The mixture was then held at 80°C for 60 minutes before 50% sodium hydroxide solution (3.15 mol) was charged maintaining temperature at 80°C. The resin was condensed at 80°C to a viscosity of 400cP.

#### EXAMPLE 2

##### Resin B: Preparation of a highly alkaline resole

Resin B is a higher alkaline phenol-formaldehyde resin than resin A, with a formaldehyde to phenol molar ratio of 2.0:1 and a sodium hydroxide to phenol molar ratio of 0.85:1. Phenol (5.0 mol) and sodium hydroxide (0.1

mol) were charged to a reaction vessel and the temperature maintained at 65°C whilst 50% formalin (3.0 mol) was added. The temperature was allowed be raised to 80°C and maintained at 80°C while a second charge of 50% formalin (5.0 mol) was added slowly over 30 minutes. The mixture was then held at 80°C for 60 minutes before 50% sodium hydroxide solution (4.15 mol) was charged maintaining temperature at 80°C. The resin was condensed at 80°C to a viscosity of 250cP.

### EXAMPLE 3

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Resin C: Preparation of a neutral resole

Phenol (1 mol) and sodium hydroxide (0.004 mol) were charged to a reaction vessel and the temperature maintained at 50°C whilst 50% formalin (0.6 mol) was added. The temperature was then raised to 80°C. The temperature was maintained at 80°C as a second charge of 50% formalin (1.0 mol) was added slowly over 30 minutes. The mixture was then held at 80°C for a further 45 minutes. The pH was adjusted with p-toluene sulphonic acid solution to 6.0 +/- 0.2. The resin was cooled to 60°C and then dehydrated by vacuum distillation until a viscosity of 200 cP was reached. The resulting resin had a resin solids content of 72%.

### EXAMPLE 4

An ester-cured resole resin for use as an electrolyte was prepared by mixing 50g of resin A with 10g of butyrolactone in a paper cup. Part of the mixture was poured into a latex mould and allowed to harden. A gel time was recorded from the mixture left in the cup.

30

### EXAMPLES 5-10

In examples 6-10, the same procedure as in example 5 was followed to prepare further ester-cured resole resins from the ingredients set out in the following Table 1:

TABLE 1

Example	Ingredient
6	50g of resin B was mixed with 12g of butyrolactone
7	50g of resin C was cooled to below 10 °C (to prevent exotherm) and mixed with 2g of acid
8	50g of resin A was mixed with 10g of triacetin
9	50g of resin A was mixed with 10g of PEG 400 and then 10g of butyrolactone
10	50g of resin A was mixed with 50g of graphite and 10g of butyrolactone

The cast specimens obtained from Examples 5 to 10 were allowed to stand at room temperature over 24 hours before conductivity measurements were made using a Como DT3800 Digital Multimeter.

The conductivity results are shown in Table 2.

TABLE 2

Conductivity measurements at 20°C on cast specimens prepared in Examples 5-10

Example	Gel time	Conductivity (S cm <sup>-1</sup> )
5	2 min 25 sec	$1.24 \times 10^{-4}$
6	2 min 15 sec	$1.42 \times 10^{-4}$



7	N/A	$> 5.00 \times 10^{-8}$
8	15 min	$7.14 \times 10^{-5}$
9	N/A	$2.42 \times 10^{-5}$
10	N/A	$1.12 \times 10^{-1}$

Examples 5-7 illustrate the conductivity induced by the formation of the alkali metal salts in the ester cured phenolic resole reactions (examples 5 and 6) compared to the acid cured resole composition (example 7).

5

It should be noted that the conductivity is further enhanced in example 6 by the use of a resin with a higher concentration of alkali. The quantity of salt in the cured polymer matrix is directly proportional to the alkali concentration in the resin. The concentration of alkali in the resin is only

10 limited by the mechanical requirements of the cured polymer composition. Typical salt levels can be calculated. For example, Resin A contains 10.7% sodium hydroxide. Therefore in 50g resin A there is 5.35g sodium hydroxide. In example 8, during reaction with the ester, triacetin, 11.1g sodium acetate is formed. This represents 18.5% salt in  
15 the total composition. When dehydrated the salt content will represent up to 25% of the cured dry phenolic composition. By adjusting the alkali content and selection of ester the final salt content can be increased or decreased.

20 Example 8 demonstrates the difference in hardening rates when different esters are selected. In example 8 triacetin gels resin A in 15 minutes whereas butyrolactone in example 5, gels resin A in 2 minutes and 25 seconds. This is an important processing feature. Esters can be selected on the basis of their saponification rate or alternatively fast reacting  
25 esters can be blended with slow reacting esters to give a chosen gel time. Example 9 was carried out to demonstrate that plasticisers could be incorporated into the cured polymer matrix. Cured phenolic resin

compositions are known to be rigid and inflexible. It is therefore important that a means to introduce flexibility is available. Polyethylene glycol (PEG) was used in this example but this does not exclude other plasticisers compatible with alkali for example novolak resins.

5

Graphite is a well-known conductivity promoter and is commonly used in electronic applications such as electrode compositions and antistatic products. To demonstrate the versatility of the alkaline resole resin, a quantity of graphite was mixed with the resin before addition of the curing agent. The casting prepared from the mixture exhibits good conductivity.

10

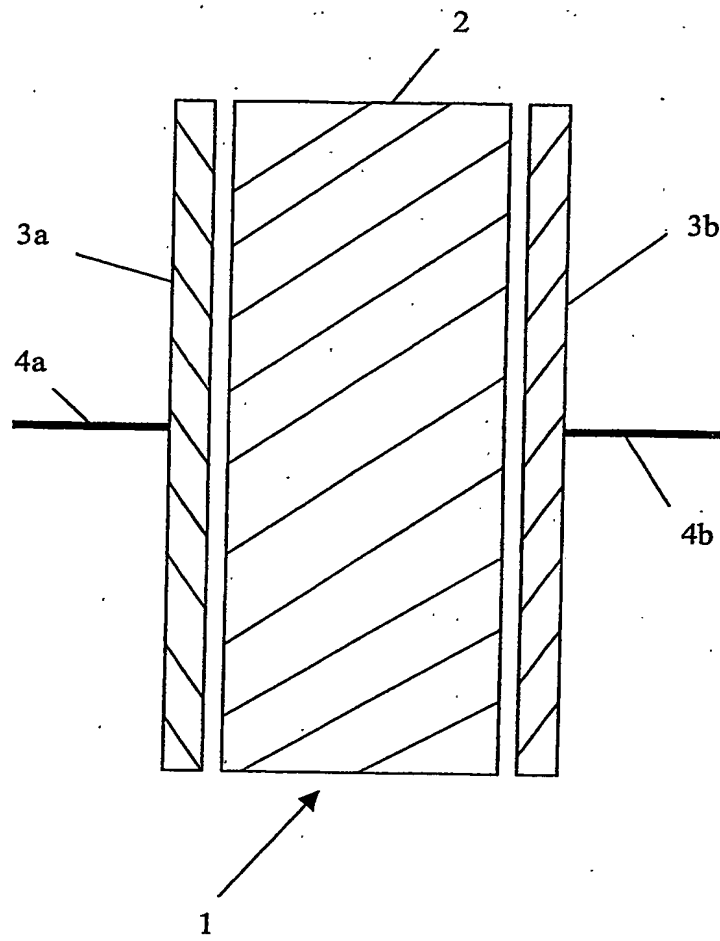


FIGURE 1

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